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SPECIFICATION FOR DI-TERT-BUTYL-PARA-CRESOL, TECHNICAL

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Gr 3 *December* **1979**

AMENDMENT NO. 1 DECEMBER 1989
TO
IS : 9207 - 1979 SPECIFICATION FOR DI-TERT-BUTYL-PARA-CRESOL, TECHNICAL

[*Page* 4, *Table* 1, *col* 3, *Sl No.* (ii)] — Substitute '68·7°C' *for* '68·5°C'.

[*Page* 6, *clause* **A-2.0**, *second sentence*] — Substitute the following for the existing sentence:

'The infra-red method shall be the referee method.'

(PCDC 9)

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Indian Standard

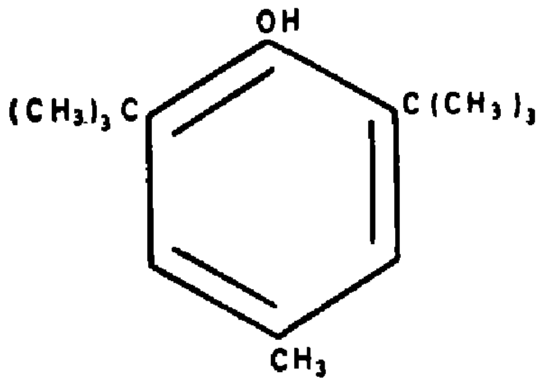
SPECIFICATION FOR
DI-TERT-BUTYL-PARA-CRESOL, TECHNICAL

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 29 June 1979, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 Di-tert-butyl-para-cresol, technical is used as an antioxidant in lubricating oils, aviation gasoline, plastics and synthetic rubbers. It also finds use in the processing of petroleum products

0.3 Chemical Name — 2,6-di-tertiarybutyl-para-cresol; 4-methyl-2,6-di-tertiarybutylphenol. Its empirical formula is $C_{15}H_{24}O$. Its molecular mass is 220·34. Structural formula of di-tert-butyl-para-cresol, technical is:



0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for di-tert-butyl-para-cresol, technical

2. REQUIREMENTS

2.1 Description — Di-tert-butyl-para-cresol, technical is a white, crystalline or flaked solid having a characteristic faint aromatic odour.

2.2 Solubility — One gram of the material shall be soluble in 4 ml of ethanol but insoluble in water. It is freely soluble in toluene, methanol, ethanol, isopropanol, ethyl methyl ketone, acetone, ether and benzene.

2.3 Identification

2.3.1 To 10 ml of a one ppm solution of the sample in methanol, add 10 ml of water, 2 ml of sodium nitrate solution (3 g in 1 000 ml), and 5 ml of dianisidine solution (200 mg of dianisidine dissolved in a mixture of 40 ml of methanol and 60 ml of 1 N hydrochloric acid). An orange red colour shall develop within 3 minutes. Add 5 ml of chloroform and shake The chloroform layer shall exhibit a purple or magenta colour which fades when exposed to light.

2.3.2 Dissolve a few crystals of di-tert-butyl-para-cresol, technical in about 10 ml of ethanol and add 0·5 ml of 0·2 percent potassium ferricyanide solution and 0·5 ml of 0 5 percent ferric sulphate in 1 N sulphuric acid. A green to blue colour shall be obtained.

2.4 The material when tested according to the methods given in Appendix A shall also comply with the requirements given in Table 1 References to the relevant clauses of Appendix A are given in col 4 of Table 1.

TABLE 1 REQUIREMENTS FOR DI-TERT-BUTYL-PARA-CRESOL, TECHNICAL

Sl No.	Characteristic	Requirement	Method of Test (Ref to Cl No. in Appendix A)
(1)	(2)	(3)	(4)
i)	Purity as $C_{15}H_{24}O$, percent by mass, *Min*	98 5	A-2
ii)	Melting point, *Min*	68·7°C	A-3
iii)	*Moisture, percent by mass, Max*	0 3	A-4
iv)	Sulphated ash, percent by mass, *Max*	0 01	A-5

3. PACKING AND MARKING

3.1 Packing — The material shall be securely packed in well-filled containers with minimum access to air and light. The containers shall be such as to preclude contamination of the contents with metals or other impurities.

3.2 Storage — The material shall be stored in a cool and dry place so as to avoid excessive exposure to heat.

3.3 Marking — Each container shall be marked legibly with the following information:

a) Name of the material,

b) Name of the manufacturer,

c) Minimum net content, and

d) Batch or code number.

3.3.1 The container may also be marked with Standard mark.

3.3.2 The use of the Standard Mark is govered by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 The representative samples of the material shall be drawn and their conformity to the standard shall be determined as prescribed in Appendix B.

APPENDIX A

(*Clause* 2.4)

ANALYSIS OF DI-TERT-BUTYL-PARA-CRESOL, TECHNICAL

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. PURITY

A-2.0 Two methods namely, infra-red and colorimetric methods have been prescribed. The infra-red method shall be the referee method.

A-2.1 Infra-red Method

A-2.1.1 *Procedure* — Weigh 0·600 0 ± 0·000 2 g of di-tert-butyl-para-cresol, technical, into a 10-ml volumetric flask, dissolve the sample in carbon disulphide and fill to the mark with this solvent. Fill a 0·2-mm liquid sample cell with the solution and measure the infrared spectrum of the sample from 11 to 14 μ using a 1·3-cm rock salt plate in the reference beam, a scan speed of 2 min/μ and a 2 × slit setting. Draw background line from 12·55 to 13·60 μ and determine the net absorption at 12·85 μ by subtracting the background absorption at this wavelength from the observed absorption. Determine the assay by referring the net absorption value to a calibration curve (*see* **A-2.1.2**).

A-2.1.2 *Preparation of Calibration Curve* — Weigh 0·48, 0·54 and 0·60 g of a purified sample of di-tert-butyl-para-cresol, technical, into three 10-ml volumetric flasks with an accuracy of ± 0·000 2 g. Dissolve the sample in carbon disulphide and dilute to the mark with this solvent. Measure the infra-red spectra of the samples from 11 to 14 μ using the same cell and the same instrumental conditions as for the sample. Determine the net absorption at 12·85 μ using the procedure described under **A-2.1.1**, and plot the value against the concentration of di-tert-butyl-para-cresol, technical (0·48 and 0·60 g samples represent 80 and 100 percent, respectively) Draw the best straight line through the three points. If the deviation of any of the points from the line is equivalent to more than 1 percent di-tert-butyl-para-cresol, prepare a fourth solution containing from 0·48 to 0·60 g of the reference sample and obtain another calibration point.

---

*Specification for water for general laboratory use (*second revision*).

A-2.2 Colorimetric Method

A-2.2.1 *Reagents*

A-2.2.1.1 *Ethanol* — absolute and 72 percent which shall be purified as follows:

> To one litre of absolute alcohol add approximately one gram of potassium hydroxide and one gram of potassium permanganate and distil in all glass apparatus discarding first 50 ml of the distillate. Prepare 72 percent alcohol (*v/v*) by diluting the purified absolute alcohol.

A-2.2.1.2 *2,2 ' - bipyridine solution* — Dissolve 0·100 g of 2,2 ' - bipyridine in 50 ml of purified absolute ethanol.

A-2.2.1.3 *Ferric chloride solution* — 4·5 percent (*m/v*) solution of ferric chloride ($FeCl_3 . 6H_2O$) in water (0 5 N).

A-2.2.2 *Procedure* — Weigh 0·100 g di-tert-butyl-para-cresol, technical into a 100-ml volumetric flask, dissolve in absolute ethanol and dilute to the mark with this solvent. Pipette 2·0 ml of the solution into a 100-ml volumetric flask and dilute to the mark with 72 percent ethanol. Pipette 5·0 ml of the diluted solution into a 25-ml glass-stoppered volumetric flask bound with black tape or metal foil to exclude the light. Add 3·0 ml of absolute ethanol, 2·0 ml of 2,2'-bipyridine solution and 2 0 ml of ferric chloride solution. Stopper the flask immediately and gently swirl the contents. Allow the solution to stand for 1 hour and measure the absorption at 515 mμ using 1·0 cm cells. The measurements are made with reference to a blank prepared at the same time as the sample and containing 5·0 ml of 72 percent ethanol, 3 0 ml of absolute ethanol and 2 0 ml of 2,2'-bipyridine solution and 2·0 ml of ferric chloride reagent. Calculate the assay by comparing the absorption obtained with the sample and that obtained with a purified specimen standard reference according to the following equation:

$$\text{di-tert-butyl-para-cresol, technical, percent by mass} = \frac{\text{Absorption of sample}}{\text{Absorption of standard specimen}} \times 100$$

NOTE — The absorption obtained with a pure sample of di-tert-butyl-para-cresol, technical, should be about 0 47.

A-3. MELTING POINT

A-3.1 Procedure — Determine the melting point according to the method prescribed in IS : 5762-1970* using the thermometer prescribed in **A-3.1.1**.

---

*Methods for determination of melting point and melting range.

A-3.1.1 *Thermometer* — of mercury-in-glass type with a range from 0 to 150°C graduated at every 0.2 deg, with partial immersion mark at 100 mm, and having a maximum error of ± 0.2 deg. A thermometer of other suitable range and accuracy may also be used.

A-4. DETERMINATION OF MOISTURE CONTENT

A-4.1 The moisture content of the material shall be determined by the procedure prescribed in IS : 2362-1973* taking about 1 g of material weighed to the nearest milligram.

A-5. DETERMINATION OF SULPHATED ASH

A-5.1 Reagents

A-5.1.1 *Concentrated Sulphuric Acid* — (*see* IS : 266-1977†).

A-5.1.2 *Dilute Nitric Acid* — approximately 0.5 N.

A-5.2 Procedure — Weigh accurately about 2 g of the material in a tared crucible. Ignite gently at first, until the material is thoroughly charred. Cool and add one or two drops of nitric acid and heat to remove the nitrous fumes. Now moisten the residue with 1 ml of sulphuric acid and ignite gently till the carbon is completely consumed. Cool the crucible in a desiccator and weigh.

NOTE — Carry out the ignition in a place protected from air currents and use as low a temperature as possible to effect the combustion of carbon.

A-5.3 Calculation

$$\text{Sulphated ash, percent by mass} = \frac{M_1}{M_2} \times 100$$

where

M_1 = mass in g of the residue, and

M_2 = mass in g of the material taken for the test.

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*Determination of water by the Karl Fischer method (*first revision*).

†Specification for sulphuric acid (*second revision*).

APPENDIX B

(*Clause* 4.1)

SAMPLING OF DI-TERT-BUTYL-PARA-CRESOL, TECHNICAL

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

B-1.2 The sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 The samples shall be placed in clean, dry, air-tight glass containers or other suitable containers on which the material has no action.

B-1.6 Each sample container shall be sealed air-tight with a stopper after filling, and marked with full details of sampling, the date of sampling, and the batch or code number.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

B-2.2 For ascertaining the conformity of the material to the requirements of the specification, samples shall be tested from each lot separately.

B-2.3 The number (n) of containers to be selected from the lot shall depend on the size (N) of the lot and shall be according to Table 2.

TABLE 2 SCALE OF SAMPLING

(*Clauses* B-2.3)

NUMBER OF CONTAINERS IN THE LOT (N)	SAMPLE SIZE (n)
(1)	(2)
2 to 15	2
16 ,, 40	3
41 ,, 65	4
66 ,, 110	7
111 and above	10

B-2.3.1 These containers shall be selected at random from the lot. In order to ensure the randomness of selection, reference may be made to IS : 4905-1968*. In case this standard is not readily available, following procedure may be adopted:

> Starting from any container in the lot, count them in one order as 1, 2, 3, ..., up to *r* and so on where *r* is the integral part of *N*/*n* Every *r*th container thus counted shall be withdrawn from the lot to give the requisite number of containers for test.

B-3. TEST SAMPLES AND REFEREE SAMPLE

B-3.1 Draw with an appropriate sampling instrument small but approximately equal quantities of the material from each container selected according to **B-2.3** after thoroughly shaking. Mix all the portions so drawn thoroughly, to form a composite sample of the quantity sufficient to make triplicate determination for all the characteristics given in this specification. Divide the composite sample into three equal parts. Each part thus obtained shall constitute a test sample and shall be sufficient to conduct all the tests. The test samples shall be transferred immediately to thoroughly clean and dry containers and sealed air-tight. These shall be labelled with the particulars given in **B-1.6**. One test sample shall be for the purchaser and another for the vendor.

B-3.2 Referee Sample — The third sample bearing the seals of the purchaser and the vendor shall constitute the referee sample to be used in case of a dispute between the purchaser and the supplier and shall be kept at a place agreed to between the two.

---

*Methods for random sampling.

B-4. NUMBER OF TESTS AND CRITERIA FOR CONFORMITY

B-4.1 All the characteristics given in the specification shall be tested on the composite sample.

B-4.2 The lot shall be declared as conforming to the requirements of the specification, if all the test results on the composite samples satisfy the relevant requirements as specified.

(*Continued from page* 2)

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